(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
15 May 2003 (15.05.2003)

PCT

(10) International Publication Number
WO 03/040243 A1

(51) International Patent Classification[7]: C09D 5/34, 7/12, C08K 7/28, C04B 26/02

(21) International Application Number: PCT/GB02/05059

(22) International Filing Date:
8 November 2002 (08.11.2002)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
0126870.5 8 November 2001 (08.11.2001) GB

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(81) Designated States *(national)*: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW.

(84) Designated States *(regional)*: ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:
— *with international search report*
— *before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

WO 03/040243 A1

(54) Title: FILLING COMPOSITION CONTAINING HOLLOW GLASS SPHERES

(57) Abstract: A filling composition e.g. for filling cracks or other defects in walls, ceilings etc, comprises an admixture of 15-40% by weight of "primary" microspheres having a density of 0.3 to 0.4 g cc^{-1} and a resin system for effecting hardening of the applied composition. The composition is lightweight and, when hardened, is 'self plugging' in the sense that a screw may be driven into the composition without the need for a separate plug.

FILLING COMPOSITION CONTAINING HOLLOW GLASS SPHERES

The present invention relates to filling compositions intended particularly, but not exclusively, for use in "DIY" repair operations such as the filling of cracks, holes pilts and other defects in walls, ceiling or the like.

Numerous filling composition are available on the market for use in household "repair" operations, such as the filling of cracks etc. in surfaces (walls, ceilings etc.) that are subsequently to be decorated by the application of paint or wallpaper. For convenience many such commercial filling compositions are sold (in a lidded tub or the like) in a "ready-to-apply" form which is akin to a stiff paste. However once applied to the surface to be repaired, the filling composition hardens so as to repair the surface. "Ready-to-apply" filling compositions of this type are manufactured by the present applicants and sold under the name TETRION (Registered Trade Mark).

Generally the "ready-to-apply" filling compositions comprise a resin/binder system (capable of effecting hardening of the composition) by loss of water, a filler and other ingredients such as one or more of a surfactant, rheology modifier, pigment and biocide.

The amount and type of the filler used determines the density and certain other characteristics of the filling compositions. Thus one such conventional composition contains about 70% by weight of limestone as the filler and has a specific gravity of 1.7-2.0. Such a composition has sufficient strength, when hardened, to be drilled (to allow the insertion of a plug for fixing a screw) or to have a nail driven therein, although they do tend to crack if a screw is fitted directly (i.e. without the use of a plug) in the hardened compositions). There is however a disadvantage of such compositions in that, due to their specific gravity, they tend to "sag" when applied to a vertical surface or a ceiling. This problem of "sag" is overcome in so-called "lightweight" filling compositions which generally have a specific gravity of about 0.35 achieved by the use of a hollow glass microspheres as the filler. However such

"lightweight" filling compositions do not have sufficient strength to be drilled or nailed.

It is an object of the present invention to obviate or mitigate the abovementioned disadvantages.

According to the present invention there is provided a filling composition comprising an admixture of 15-40% by weight of "primary" microspheres having a density of 0.3 to 0.4 g cc^{-1} and a resin system for effecting hardening of the applied composition.

Compositions in accordance with the invention may have a specific gravity of 0.4 to 0.5 and as such are not prone to "sagging" during application, e.g. to a vertical surface or a ceiling. Nevertheless we have surprisingly found that the inclusion of microspheres as defined in the previous paragraph imparts strength to the composition to the extent that (even with the relatively low specific gravity) it may (when hardened) be drilled or a nail driven therein. Additionally we have also found that the hardened filler is "self-plugging" in the sense that a screw may be driven into the hardened composition without the need for a separate plug, although drilling of a pilot hole will be advantageous.

Preferably the composition comprises 15-35%, more preferably 20 to 30%, even more preferably 23 to 27% and ideally about 25% by weight of the "primary" microspheres.

The "primary" microspheres are preferably hollow and are preferably of glass (e.g. soda lime borosilicate glass).

It is preferred that the "primary" microspheres have a density in the range 0.35 to 0.39, e.g. about 0.37 g cc^{-1}.

A further preferred feature is that the "primary" microspheres have a particle size of less than 90μm, preferably such that 50% by volume of the "primary" microspheres have a size less than a value in the range 35 to 45μm (e.g.40μm) and 50% by volume have a size above that value.

Preferably also the "primary" microspheres have a crush strength (90% survival) of at least 2000psi, more preferably 2000-4000psi, even more preferably 2500-3500psi, still more preferably 2800-3200psi and most preferably about 3000psi.

Suitable glass microspheres for use as the "primary" microspheres are available from Zeelan Industries, Inc. (a wholly owned subsidiary of 3M) under the designation "3M Scotchlite Glass Bubbles", the grade K37 being particularly suitable.

Preferred embodiments of filling composition in accordance with the invention also incorporate 5 to 15%, preferably 8 to 12%, by weight of "secondary" microspheres having a density of 0.15 to 0.25 g cc^{-1}. Such "secondary" microspheres do not of themselves impart significant strength to the composition but nevertheless may be incorporated to provide volume thus permitting adjustment of the specific gravity of the composition. Suitable "secondary" ceramic microspheres are similar to the "primary" microspheres to the extent that they are preferably hollow and preferably of glass (e.g. soda lime borosilicate glass).

It is preferred that the "secondary" microspheres have a density of 0.18 to 0.22 g cc^{-1} (preferably about 0.20 g cc^{-1}). The "secondary" microspheres preferably have a size in the range 10 to 120 μm and are preferably such that 50% by volume are of the size less than a value in the range 60 to 70μm (preferably 65μm) and 50% by volume are of a larger size.

The "secondary" microspheres preferably have a crush strength (90% survival) in the range 300 to 800, more preferably 400 to 700 and ideally about 500psi.

Suitable glass microspheres for use as the "secondary" microspheres are available under the designation " 3M Scotchlite Glass Bubble", the grade K20 being particularly suitable.

A preferred composition in accordance with the invention incorporates 23 to 27% (preferably 25%) by weight of Scotchlite K37 microspheres and 7-10%, e.g. about 8.5%, of Scotchlite K20 Glass Microspheres.

It is preferred that filling composition of the invention contains 20 to 30% by weight of the resin which also acts as a binder for the composition. Resin is preferably a homopolymer or copolymer of unsaturated (preferably olefinically unsaturated) monomers. A copolymer of styrene and an acrylic acid ester is preferred.

The resin system for the filling composition may be provided as a water-based dispersion or emulsion of the homopolymer or copolymer which effects hardening on loss f water (e.g. by evaporation) once the composition has been applied. The dispersion/emulsion may be surfactant stabilised and (prior to admixture with the other components of the filling composition) will generally have a solids content of about 50% by weight. Typically the amount of the emulsion incorporated in the formulation will be 50-55% by weight (giving a resin content in the composition of about 25-26%). A particular suitable dispersion is available under the name REVACRYL 385 (ex HARCO) which is a 50% dispersion in water of a copolymer of styrene and acrylic acid ester.

The composition may include additional filler (other than ceramic microspheres), e.g. in an amount of 5-15% by weight. A particularly preferred additional filler is dolomite which also serves to impart "whiteness" to the composition. However other lime stone fillers could be used if "whiteness" is not particularly required.

The composition may incorporate minor amounts of other components to provide the required final properties for the composition.

Thus, for example, the composition may incorporate a thickener/rheology, such as an acrylic copolymer initially provided in the form of an aqueous emulsion containing anionic surfactant. The amount of the copolymer incorporated in the composition may, for example, be 0.1-2%. The thickener may be provided by VISCALEX PL30 (ex CIBA SPECIALITY CHEMICALS INC) which is a 30% aqueous emulsion of an acrylic co polymer and containing also an anionic surfactant.

Other rheology modifiers that could be included are laponite (e.g. used in an amount of 1-2%) and/or poly(ethylene glycol) such as PEG 600 used in an amount of 0.1-2%.

The composition will with advantage contain a coalescing solvent which assists hardening of the resin system. A suitable coalescing solvent is diethylene glycol monobutyl ether, e.g. used in an amount up to 0.25% by weight.

An agent for assisting dispersion of the filler materials in the composition will with advantage also be included. A particular suitable dispersing additive is DISPERBYK 190 which is a solution of high molecular weight block copolymer with pigment affinic groups.

Additional additives include biocides (e.g. up to 0.5% by weight) such as isothizolinones, e.g. one or more of 5-chlro-2-methyl-4-isothizolain-3-one, 2-methyl-4-isothiozolain-3-one and 2-n-octyl-4-isothiozolain-3-one.

Ingredients such as anti-foam agents may be included in the manufacturing process.

The invention is described with reference to the following non-limiting example.

Example 1

	Component	Amount (% by weight)
a	Revacryl 385	52.5
b	Acticide BX	0.25
c	Acticide DW	0.05
d	Disoerbyk 190	0.6
e	Butyl Di Glycol	0.1
f	Viscalex PL30	1.6
g	Microdol H150	11.6
h	Scotchlite K37	25.00
i	Scotchlite K20	8.5

Using a vacuum process mixer (Molteni Pot), components (a)-(e) were mixed for 10 minutes until homogeneous. Component (f) was slowly added and mixing continued for 10 minutes to allow the composition to thicken. Next component (g) was added slowly, allowing the powder to wet out and mixing continued for 10 minutes. Finally components (a) and (i) were added slowly, allowing the powder to wet out.

The product was a white, soft smooth paste having an S.G (Gallon Cup) in the range 0.42-0.5 and a pH in the range 8.2-9.0. The viscosity (RVT) was in the range 25-40.

The composition was self-hardening when applied to a surface and did not demonstrate a tendency to sag.

A filling composition was prepared from the following components:

CLAIMS

1. A filling composition comprising an admixture of 15-40% by weight of "primary" microspheres having a density of 0.3 to 0.4 g cc^{-1} and a resin system for effecting hardening of the applied composition.

2. A composition as claimed in claim 1 comprising 20 to 30% by weight of the "primary" microspheres.

3. A composition as claimed in claim 2 comprising 23 to 27% by weight of the "primary" microspheres.

4. A composition as claimed in any one of claim 1 to 3 wherein the "primary" microspheres have a density in the range 0.35 to 0.39 g cc^{-1}.

5. A composition as claimed in any one of claims 1 to 4 wherein the "primary" microspheres have a particle size of less than 90μm.

6. A composition as claimed in any one of claim 1 to 5 wherein 50% by volume of the "primary" microspheres have size less than a value in the range 35 to 45μm and 50% by volume have a size above that value.

7. A composition as claimed in any one of claims 1 to 6 wherein the "primary" microspheres have a crush strength (90% survival) of 2800-3200 psi.

8. A composition as claimed in any one of claim 1 to 7 wherein the "primary" microspheres are hollow.

9. A composition as claimed in any one of claims 1 to 8 wherein the "primary" microspheres are of glass.

10. A composition as claimed in any one of claims 1 to 9 incorporating 5 to 15% by weight of "secondary" microspheres having a density of 0.15 to 0.25 g cc^{-1}.

11. A composition as claimed in claim 10 containing 8 to 12% by weight of the "secondary" microspheres.

12. A composition as claimed in claim 10 or 11 wherein the "secondary" microspheres have a density of 0.18 to 0.22 g cc^{-1}.

13. A composition as claimed in any one of claims 10 to 12 wherein the "secondary" microspheres have a size in the range 10 to 120 μm and are such that 50% by volume are of a size less than a value range 60 to 70 μm and 50% by volume are of a size above that value.

14. A composition as claimed in any one of claims 10 to 13 wherein the "secondary" microspheres have a crush strength (90% survival) in the range 400 to 700 psi.

15. A composition as claimed in any one of claims 10 to 14 wherein the "secondary" microspheres are hollow.

16. A composition as claimed in any of claims 10 to 15 wherein the "secondary" microspheres are of glass.

17. A composition as claimed in any one of claims 1 to 16 wherein the resin a copolymer of unsaturated monomers.

18. A composition as claimed in claim 17 wherein the resin is a copolymer of styrene and acrylic acid ester.

19. A filling composition comprising:

(i) 23 to 27% by weight of "primary" microspheres having a density of 0.35 to 0.39 g cc^{-1} and a crush strength (90% survival) of 2800-3200psi;

(ii) 8 to 12% by weight of "secondary" microspheres having a density of 0.18 to 0.22 g cc^{-1} and a crush strength (90% survival) in the range 400 to 700 psi; and

(iii) a resin system for effecting hardening of the applied composition.

20. A filling composition substantially as herein before described in Example 1.

INTERNATIONAL SEARCH REPORT

PCT/GB 02/05059

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C09D5/34 C09D7/12 C08K7/28 C04B26/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C09D C08K C09K C04B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

* Special categories of cited documents :

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Date of the actual completion of the international search	Date of mailing of the international search report
26 February 2003	26/03/2003

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